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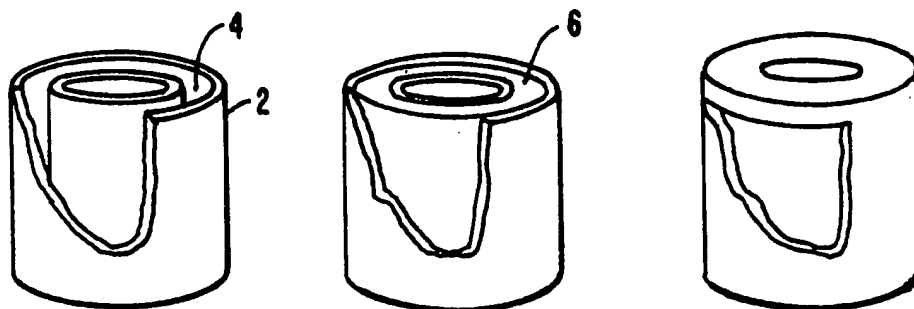
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(54) Title: SEALS WITH LOW THERMAL EXPANSION



(57) Abstract

An improved sealing system. The sealing system provides for the use of a body (2) defining a cavity (4). The cavity is filled or substantially filled with a material (6) having a lower coefficient of thermal expansion than the body and, in some embodiments, a material that expands at low temperature. During low temperature operation, the body may shrink considerably, but the compressive force applied between two surfaces will not decrease appreciably due to the internal, low coefficient of thermal expansion filler. Accordingly, the risk of seal failure at low temperature is substantially reduced.

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SEALS WITH LOW THERMAL EXPANSION

BACKGROUND OF THE INVENTION

The present invention relates to the field
5 of sealing systems and methods of forming seals. For
instance, in one embodiment the invention provides a
seal system that is resistant to leakage due to low
temperatures, as well as a method of using such sealing
systems and methods of their manufacture.

10 Gaskets and other sealing members are used in
a broad array of applications to prevent encroachment of
material beyond two adjoining surfaces. Such gaskets are
normally placed between the two surfaces and the two
surfaces are brought together, compressing the gasket
15 between the two surfaces. Accordingly, fluids, gases and
other materials are prevented from flowing between the
two surfaces. Such gaskets have been made from many
different materials ranging from cork to exotic
elastomers. Representative gaskets are described in
20 U.S. Patent Nos. 3,524,794, 4,196,162, 4,317,575, and
2,806,509.

Often gaskets are subjected to low temperatures
during their life cycle. Although the gasket may seal
satisfactorily at its installation temperature, as the
25 temperature decreases the size of the seal decreases.
Therefore, a decrease in the compressive force between
the surfaces abutting the gasket is often observed during
low temperature operations. When the compressive force
decreases beyond a certain level, the gasket will be
30 subject to leakage and failure. Fig. 1 illustrates
the volume change (in %, from 20°C) as a function of
temperature for various elastomers. As seen, the
volume change is significant, and this can hurt gasket
performance at low temperatures. The data therein are
35 taken from Robbins et al., Adv. Cryog. Eng. (1963)
pgs. 287-299.

While substantial work has been done to improve the life and sealing capabilities of gaskets used at high temperatures, less work has been done to solve the problems of gaskets operated at low temperatures.

5 In large part, such efforts have been directed at different materials such as those described in U.S. Patent Nos. 5,093,432, 4,981,727, 4,673,187, and 4,580,794. While meeting with some success, such gasket materials have continued to be subject to failure,
10 especially during low temperature operations.

From the above it is seen that improved methods and articles for gasket formation are needed.

SUMMARY OF THE INVENTION

15 An improved sealing system is provided by virtue of the present invention. The sealing system provides for the use of a body defining a cavity. The cavity is filled or substantially filled with a material having a lower coefficient of thermal expansion than the
20 body or materials that expand at low temperatures (e.g. water). It is to be understood that the term "lower coefficient of thermal expansion" includes materials that may expand over at least a portion of a low temperature range of interest. In most cases herein the temperature
25 range of interest will be below ambient such as below about 4°C, and often in the range of 20°C to -40°C.

During low temperature operation, the body may shrink or attempt to shrink considerably due to its large coefficient of thermal expansion, but the compressive
30 force applied between two sealed surfaces will not decrease appreciably due to the internal, low coefficient of thermal expansion filler. The low coefficient of thermal expansion filler prevents excessive contraction of the seal. Accordingly, the risk of failure at low
35 temperature is substantially reduced.

The seals according to the present invention are easily installed, and highly conformable. The seals

require little compression to seal, and have minimal change in dimensions as temperature is changed. The seals may be resistant to chemical and environmental attack, through appropriate selection of the body material while also being easily reusable. Conversely, the filler material may be selected from a wide range of materials that may or may not be resistant to environmental attack.

A further understanding of the nature and advantages of the inventions herein may be realized by reference to the remaining portions of the specification and the attached drawings.

BRIEF DESCRIPTION OF THE DRAWINGS

Fig. 1 is an illustration of the coefficient of thermal expansion of various materials;

Figs. 2a to 2c illustrate the manufacture of one embodiment of a gasket herein;

Figs. 3a to 3c illustrate compressive force as a function of temperature;

Fig. 4 illustrates a cable enclosure assembly;

Figs. 5, 6, and 7 illustrate compressive force for water based seals;

Figs. 8a, 8b, and 8c illustrate an alternative embodiment of the seal;

Figs. 9a, 9b, 9c, and 9d illustrate a cable enclosure, along with various seals therefore;

Figs. 10a and 10b illustrate an in-line cable enclosure;

Figs. 11a and 11b illustrate a butt splice enclosure;

Figs. 12a, 12b, 13a, and 13b illustrate seals with soft rings;

Figs. 14a and 14b illustrate seals with compression rings;

Figs 15a to 15c illustrate a dual seal system;

Figs. 16a to 16d illustrate another version of a dual seal system;

Figs. 17a to 17c illustrate another version of a dual seal system;

5 Figs. 18a to 18c illustrate another version of a dual seal system; and

Figs. 19a to 19d illustrate another version of a dual seal system.

10 DESCRIPTION OF THE PREFERRED EMBODIMENTS

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I. General

30 Figs. 2a to 2c illustrate one embodiment of the invention, along with a method of making such embodiments. As shown, the invention provides for the use of a body 2. The body defines an internal space 4. In the particular embodiment shown in Fig. 2, the body is
35 cylindrical in shape, although the shape of the body will vary dramatically from application to application depending upon the shape of the surfaces to be sealed.

The body is preferably an elastomeric material, i.e., a material that at least partially recovers its original shape when compressed or stretched.

As shown, the body is initially open on its top surface. The entire top surface may be fully open as shown in Fig. 2, or one or more small apertures may be provided in the body. The opening(s) in the top of the body are used to place a fill material 6 in the body as shown in Fig. 2b. The internal space 4 is substantially filled with the fill material such that the fill material is closely packed therein. As used herein, the term "substantially filled" is intended to mean filling of more than 30% of the volume of the internal space with the fill material, preferably more than 50% of the internal space, more preferably more than 70% of the internal space, preferably more than 90% of the internal space, preferably more than 95% of the internal space, and most preferably more than 99% of the volume of the internal space.

The body and the fill material have different coefficients of thermal expansion, the fill material preferably having a lower coefficient of thermal expansion than the body. By "lower", it is intended to mean herein not only materials that have lower coefficients of normal expansion, but also those that expand as temperature is decreased. Preferably, the coefficient of thermal expansion of the fill material is less than 200, preferably less than 150, preferably less than 100, preferably less than 60, preferably less than 10, and most preferably less than 5×10^{-6} m/m/°C. Preferably, the coefficient of thermal expansion of the fill material is more than 10% less than that of the body, preferably more than 50% less than the body, and most preferably more than 100% less than that of the body material.

The fill and the body materials desirably selected such that the overall structure of the seal is

both compliant and resilient. Preferred materials for the body are thermoset elastomers such as natural rubber, styrene-butadiene rubbers, butyl rubber, ethylene-propylene rubbers, polyisoprene, polybutadiene, nitrile elastomers, neoprene, polysulfide polymers, polyacrylic rubber, silicone rubbers, chlorosulfonated polyethylene, fluorocarbon elastomers, fluorosilicones, polyurethane elastomers, styrene-butadiene copolymers, epichlorohydrin polymers, phosphonitrilic fluorelastomer, tetrafluoroethylene-propylene copolymers, ethylene-methyl acrylate copolymer, or perfluoro elastomers, as well as mixtures thereof. In some embodiments the body is made of thermoplastic elastomers such as styrene-diene-styrene triblock copolymers (SBS, SIS, SEBS, SEPS), thermoplastic polyurethanes (TPU), thermoplastic olefins (TPO), elastomers/thermoplastics alloys (e.g., acrylonitrile-butadiene rubber/pvc alloy), as well as mixtures thereof. According to one specific aspect of the invention the body is RTV silicone rubber such as Silastic J available from Dow Corning or thermoplastic elastomers such as Kraton rubber.

According to some embodiments, all or part of the body is lined with a water resistant barrier such as metal foil. Such barriers may cover only a portion of the gasket such as a ring on the top of the gasket, or may line the entire inner surface of the body.

Preferred materials for the fill material include glasses, especially with a low (e.g., less than about $200 \times 10^{-6} \text{ } ^\circ\text{C}^{-1}$) coefficient of thermal expansion such as sodium borosilicate, lime magnesia aluminosilicate, 96% silica, and similar glasses; graphite or carbon pellets; ceramics; filled and unfilled thermoset and thermoplastic materials epoxys with a low (e.g., less than $100 \times 10^{-6} \text{ } ^\circ\text{C}^{-1}$) coefficients of thermal expansion; metal spheres having a low coefficient of thermal expansion, glasses, ceramics, metal and metal alloys, graphite, carbon, mica, other mineral and synthetic

fillers, filled and unfilled thermoset and thermoplastic materials, and the like, as well as mixtures thereof. The coefficient of thermal expansion of various representative materials that may be used in conjunction with the invention herein are illustrated in Table 1.

Table 1
Representative Fill Materials'
Coefficients of Thermal Expansion

	<u>Material</u>	<u>CTE</u> <u>10⁻⁶m/m/°K</u>
	Mica	0.10
15	Graphite	0.11
	Fuse	0.55
	Carbon	2.70
	Glass, Borosilicate	3
	Epoxy Resin, Ai-Filled	5
20	Aluminum Oxide	8
	Nylon, 30% Glass	15
	Copper	17
	Aluminum	25
	Poly(phenylene Oxide)	30
25	Water	52.7
	Polycarbonate	57
	Polystyrene	65
	Poly(ethylene Terephthalate)	65
	Nylon 6/6	80
30	Polypropylene	80
	Polytetrafluoroethylene	95
	Polyethylene, High Density	120
	Polyethylene, Low Density	200

Many preferred fill materials with low coefficients of thermal expansion are hard; accordingly solid blocks of such materials are often not desirable since the compliancy of the seal will be compromised. In preferred embodiments, compliancy of the seal is maintained by using finely divided particles of such fill materials without interspersed rubber or other body materials. Preferably, such non-compliant fill materials are provided with particle sizes of preferably less than

about 1000 microns, preferably less than 500 microns, and preferably less than 300 microns. According to one specific embodiment of the invention the fill material is glass microspheres having a diameter of between about 150
5 and 250 microns. Such spheres are available from Potters Industries Inc. under the name Spheriglass. The particles of fill material preferably maintain greater compliancy by leaving the interstices between the particles devoid of elastomer such as the elastomer of
10 the body. Accordingly, the particles of fill move about freely in the body, allowing the structure to be compliant. Preferably, the particles or spheres are substantially solid, i.e., each of the spheres is made of a substantially solid material such as glass. Hollow
15 particles may be used to reduce weight. In some embodiments, the void between particles of fill material is not filled. Accordingly, preferably only about 50% of the volume of the body is filled with fill material, preferably more than 75%, and more preferably more than
20 90% of filled volume will be highly dependent on particle size selection.

After filling the internal space with the fill material, the opening(s) in the body are sealed as shown in Fig. 2c. In alternative embodiments the body is
25 sealed ab initio, and the fill is injected with, for example, an injection needle, optionally with another needle for pressure venting. The resulting body and internal fill material are formed in the shape necessary to mate with adjoining surfaces that are to be sealed.

30 Gaskets made according to the invention will have a variety of uses, including conventional uses such as pipe gaskets and the like. According to one aspect of the invention the gaskets are used as seals for CATV, telephone and power cable connectors, closures for free-
35 breathing aerial applications (i.e., Raychem's TRAC® line of products, fiber optic splice closures, cable TV splice cases, pressurized and unpressurized copper cable

closures, pedestal closure system, enclosures for preconnectorized cables, and terminal boxes.

Incorporation of low CTE materials in gels, mastics, liquids, sealants and encapsulants will reduce the overall CTE of the resulting blend. These, in turn, can be encapsulated in an elastomeric sleeve).

A. Example

A gasket in which a thermocouple was embedded, was placed in a cylindrical stainless steel holder having approximately the same OD as the gasket. The assembly was placed between the arms of an Instron which were enclosed within an insulated chamber conditioned to a predetermined temperature and the gasket compressed with a given force (e.g., 100 lbs). The change in compressive force as the sample was cooled or heated from room temperature was measured and recorded. The silicone rubber gasket was made from Silastic J available from Dow Corning. The body of the comparison gasket was made of the same material. The internal space in the comparison gasket was filled with glass microspheres having an average diameter of 219 microns and available from Potters Industries.

Figs. 3a and 3b compare the performance of the silicone rubber gasket and the gasket according to the present invention. Specifically, Figs. 3a and 3b plot the compressive force for each of the gaskets as a function of temperature. Fig. 3a illustrates the results for gaskets with a tight fit (i.e., a gasket in which the outside diameter of the gasket was the same as the inside diameter of the cylinder). Fig. 3b illustrates the results for gaskets with a loose fit (i.e., a gasket in which the outside diameter of the gasket was 1.25" while the inside diameter of the cylinder was 1.351").

As shown therein, the silicone rubber gasket at room temperature has approximately the same compressive force as the gasket according to the present invention.

However, in both cases, as the temperature is lowered the compressive force exerted by the silicone rubber gasket drops much more quickly than the gasket according to the present invention. As seen in Fig. 3a, the solid rubber
5 gasket drops well below this value at about 15°C, while the gasket according to the present invention did not reach the critical value at the lowest tested temperature of -40°C. While the loose fitting silicone rubber gasket did not drop below the critical value, its compressive
10 force was substantially lower than that of the present invention.

Fig. 3c illustrates the radial deflection of a gasket as a function of compressive force applied to the gasket for both solid Silastic "J" gaskets (i.e.,
15 silicone rubber) and gaskets as described above. As shown, the gaskets according to the present invention deflect radially much more easily as pressure is applied. The results shown therein are for a gasket of 1.25" diameter and 1" height. The ability to deflect with low
20 applied force is one of the key differences between the seals herein and solid rubber seals. This allows the seals to conform around irregular shapes (conformability), to seal cables of a wide range of diameters (cable range taking ability) and allows the
25 manufacture of large size seals. Because of the high modulus of solid rubber seals, conformability, range taking ability and seal size are limited. Furthermore, the force required to effect sealing is much lower for the seals therein than for solid rubber seals.

30

B. Example

To determine the effectiveness of the invention during thermal cycling, gasket structures were applied to an aluminum test fixture as illustrated in Fig. 4. As
35 shown therein, a tubular body 8 was sealed at one end with a gasket 10 against a washer 12 and a cap 14. The gasket 10 sealed around a cable 16 that extended into the

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body 8. The other end of the device was sealed with a cap having a pressurization source/valve 18. The device was initially pressurized to 5 psi and the temperature was repeatedly cycled from -40°C to 60°C.

5 Tables 2a and 2b illustrate the results of the thermal cycling experiment. By contrast, solid silastic J gaskets failed after 12 and 23 cycles.

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Table 2aThermal Cycling Performance of Vitrosealed
(Aluminum fixtures and rods)

<u>Sample</u>	<u>Type of Fixture</u>	<u>Cable/Rod Type</u>	<u>No. of Cycles</u>	<u>Type of Failure</u>
13425-8-1	2" Lexan	.5" Al rod	305 (ongoing)	
13425-13-10	2" Al	.5" Al rod	15 (failed)	Slow leak at outer threads Molding imperfection
13425-15-11	2" Al	.5" Al rod	410 (ongoing)	
13425-15-12	2" Al	.5" Al rod	381 (ongoing)	
13425-15-13	2" Al	.4" Brass	381 (ongoing)	
13425-15-15	2" Al	.5" Al rod	54 (failed)	Slow leak at inner threads Molding imperfection
13525-48-1	2" Al	.5" Al rod	186 (ongoing)	
13525-48-2	2" Al	.5" Al rod	186 (ongoing)	
13445-35-20	2" Al	.5" Al rod	93 (failed)	Leak around rod Air void in seal
13445-35-21	2" Al	.5" Al rod	465 (ongoing)	
13445-35-22	2" Al	.5" Al rod	60 (failed)	Leak around rod Air void in seal
13445-35-23	2" Al	.5" Al rod	321 (ongoing)	

Table 2b
Thermal Cycling of Vitroseals
(Aluminum fixtures, FO Cable)

<u>Sample</u>	<u>Type of Fixture</u>	<u>Cable/Rod Type</u>	<u>No. of Cycles</u>	<u>Type of Failure</u>
13445-35-26	2" A1	.5" FO cable	123 (ongoing)	
13445-35-28	2" A1	.5" FO cable	30 (failed)	Deep scratch on FO cable
13445-35-29	2" A1	.5" FO cable	78 (ongoing)	
13445-35-30	2" A1	.5" FO cable	60 (failed)	Deep scratch on FO cable

II. First Alternative Embodiment

- According to one alternative embodiment of the invention the fill material expands at low temperatures such as below 4°C. According to such
- 5 embodiments, the body is formed in a manner similar to that of the above embodiment. Hollow, cylindrical containers are molded using, in the case of a silicone rubber, material such as Dow Corning Silastic J. The body is then filled with materials that expand at low
- 10 temperatures such as hydrogels or water-swellaable polymers. If necessary, the consistency of the gel is adjusted, and the top of the container is sealed by addition of silicone rubber mix on the top of the container.
- 15 In the case of thermoplastic elastomer bodies, the body is compression molded of, for example, Kraton rubber. The body is filled with water, hydrogels, or water swellaable polymers. The top of the container is closed by bonding disks cut from a slab of the same
- 20 rubber material as the rest of the body with a toluene solution containing the rubber. Alternatively, the structure is formed in a closed manner and the fill is added with a syringe or similar instrument.
- In this embodiment the fill material is
- 25 preferably selected from water or aqueous solutions, water absorbent materials, water swellaable polymers, natural, or synthetic hydrogels. Water solutions or dispersions preferably contain organic or inorganic compounds used as stabilizers, fungicides, thickeners,
- 30 or the like. Natural hydrogels are preferably contain materials selected from the group of gelatin, gum tragacanth, locust bean gum, guar gum, sodium alginate, gelatinous polysaccharides, and the like, as well as mixtures thereof.
- 35 In the case of synthetic hydrogels, such fill materials are preferably based on polyethylene oxide, polyacrylic and polymethacrylic acids, polyacrylic and

polymethacrylic esters, polyhydroxyalkyl metacrylates or acrylates, polyacrylamide or polymetacrylamide, poly-n-isopropylacrylamide, poly-n-vinyl-1-2-pyrrolidinone, crosslinked polyvinyl alcohol, polyvinyl propanol, polyethylene oxide, carboxymethyl or hydroxyethyl cellulose, hydroxypropyl methylcellulose, crosslinked polypropenoic acid, polyelectrolyte complexes, charged polymers carrying ionizable groups, and mixtures thereof.

10 A. Example

Tubular gaskets similar to those described above were formed from silicone rubber with their cores filled with gelatine gel (3.5% gelatine and 96.5% water). The structures were applied to an aluminum test fixture as illustrated in Fig. 4. The device was initially pressurized to 5 psi and the temperature was cycled from -40°C to 60°C.

Using a gelatine-filled gasket, the pressure in the body was maintained at 5 psi for 46 cycles. By contrast, when the same experiment was conducted using a silicone gel filled gasket the pressure began to fall after 2 cycles. Similarly, a 100% silicone rubber gasket also failed after 2 temperature cycles, demonstrating the low temperature effectiveness of the present invention.

25

B. Example

The compressive force as a function of temperature was determined using the techniques described above for both silicone gel gaskets and gaskets according to the present embodiment of the invention. The results are shown in Fig. 5. The fill material for the embodiment of the invention described herein was hydrogel prepared as in Example C.

As shown therein, the compressive force for the embodiment of the invention herein is dramatically higher at temperatures of less than 10°C, largely due to the

dramatic volume expansion of the water based material in the core of the gasket.

C. Example

5 Gaskets with several water-containing fill materials were prepared as follows:

- 10 1. HYDROGEL BASED ON SODIUM CARBOXYMETHYL CELLULOSE (Aqualon, CMC, Aqualon Co., Wilmington, Delaware, Bulletin VC-521)

Formulation:

	<u>MATERIAL</u>	<u>WEIGHT %</u>
	Dihydroxyaluminum sodium carbonate (DASC)	0.10
15	Potassium sorbate	0.14
	Water	92.43
	Propylene glycol	4.00
	Sodium carboxymethyl cellulose (CMC)	3.00
	Hydroxyethyl cellulose	0.15
20	Fumaric acid	<u>0.18</u>
		100.00

The gasket was prepared according to the following procedure:

- 25 a. Add the water to a mixing tank equipped with a Lightning mixer. Begin slow agitation and add the DASC and potassium sorbate.
- 30 b. Blend the CMC, hydroxyethylcellulose and fumaric acid. Add these ingredients to the propylene glycol and stir until they are dispersed (normally no more than 2 min.).
- c. Add the glycol slurry to the DASC dispersion and stir for 15 min.
- 35 d. Fill containers.
- e. Hold containers at room temperature for 48 hrs. before testing.

2. HYDROGELS BASED ON CROSSLINKED ACRYLIC ACID
POLYMERS (Carbopol Polymers, B.F. Goodrich,
Cleveland, Ohio, Bulletin IS-2)

Chemically, Carbopol resins are acrylic acid
5 polymers. Individual resins vary by molecular weight and
degree of crosslinking.

Formulation:

	<u>MATERIAL</u>	<u>WEIGHT %</u>
10	Carbopol 676	1.00
	Water	<u>99.00</u>
		100.00

The gasket was prepared according to the
15 following procedure:

- a. Add Carbopol resin slowly to the vortex of
the water being agitated. Add Carbopol
resins as early in a formulation as
possible. This allows for the longest
20 possible mixing time which helps insure
that the Carbopol resin is completely
dispersed and hydrated.
- b. Neutralize the Carbopol resin, that is,
increase the pH, as close to the end of the
25 process as possible. This allows for the
complete dispersion of the resin and other
formulation ingredients prior to the
Carbopol resin providing its highest
viscosity.
- 30 c. Fill parts with the resulting gel.

3. HYDROGEL BASED ON CROSSLINKED POLYPROPENOIC ACID (XU 40346 Super Absorbent Polymer, Dow Chemical, Midland, Michigan)

Chemically, XU 40346 is a partial sodium salt
5 of crosslinked polypropenoic acid.

Formulation:

	<u>MATERIAL</u>	<u>WEIGHT %</u>
	XU 40346	3.00
10	Water	<u>97.00</u>
		100.00

The gasket was prepared according to the
following procedure:

- 15 a. Add the water to a mixing tank equipped
with a propeller-type mixer. Begin
agitation and add the XU 40346 resin.
b. Fill parts with the resulting gel.

20

4. HYDROGELS BASED ON GELATIN
(Knox Gelatine, Inc.)

Formulation:

25

	<u>MATERIAL</u>	<u>WEIGHT %</u>
	Knox gelatin	3.70
	Water	<u>96.30</u>
		100.00

30

The gasket was prepared according to the
following procedure:

- a. Boil water and add the gelatine powder.
b. Cool mixture to about 40°C.
35 c. Fill containers with liquid mix.
d. Chill samples until gelatine is firm.

Experiments were conducted to determine the compressive force generated by the above water swelling polymers during cooling, and the results are shown in Fig. 6. In all cases, the compressive force generated by the seal is substantially higher using the invention herein than, for example, silicone rubber as the temperature is lowered. Similar results were obtained for tight fitting gaskets, and for both metal and plastic sealed surfaces.

Table 3 summarizes the results of various compressive force retention experiments. Specifically, Table 3 illustrates the compressive force retention of Hydoseal™ gaskets according to one embodiment of the invention herein at their minimum point, and compares these results to silastic J and silastic L. Results are illustrated at a number of different initial applied forces. As seen therein, the gaskets according to the invention herein consistently have much higher minimums than conventional gaskets.

Table 3

Compressive Force Retention of Hydoseal and Solid
Silicon Rubber Samples at their Minimum Point

Applied Force (lbs)	Hydoseal	Silastic "J"	Silastic "L"
<u>Tight Fit:</u>			
25	72%	0%	0%
50	72%	0%	0%
100	68%	0%	0%
<u>Loose Fit:</u>			
25	76%	---	35%
50	88%	---	16%
100	91%	44%	10%

C. Example

Fig. 7 illustrates the results of compressive force tests for various gaskets containing different ratios of rubber to Aqualon as described in the above example. When the seals are thermally cycled, two types of phenomena occur. First, the rubber sleeve expands when heated and contracts during cooling. On the other hand, the hydrogel or water-containing compound increases in volume when the temperature decreases below 4°C or when it increases above 20°C. The sum of the volume changes at any given temperature could be either positive or negative depending on the volume ratio of rubber to hydrogel used in making the seal.

To study the effect of hydrogel to rubber ratio, a series of samples were prepared which consisted of rubber sleeves having various wall thicknesses filled with different types of water-swellaable polymers. These samples which had a cylindrical shape (1.25" dia. x 1" length) were tested following the procedure outlined above.

The results, shown in Fig. 7, indicates that the compressive force retention of these samples was dramatically decreased as the amount of water-containing compound was reduced. As shown therein, a gasket made with 8% aqualon gel and 92% rubber (referring to the percentage of the body or inner space compared to the total gasket volume) exhibits a slight decrease in compressive force as the temperature is decreased from room temperature. By contrast, gaskets made with 26% aqualon gel and 38% aqualon gel show substantial increases in the compressive force as the temperature is lowered below room temperature. The minimum of the 38% curve is only a few percent lower than the value at 20°C.

III. Representative Applications

It will be understood that the present invention can be used as a seal that will take on any one of a number of mechanical configurations. Representative
5 embodiments are illustrated below.

A. Longitudinal Seam Sealing

Figs. 8a, 8b and 8c illustrate the mechanical configuration of a cable sealing gasket according to one
10 aspect of the invention. According to the embodiment shown in Fig. 2, the gasket includes a body 22 filled with a low coefficient of thermal expansion material or water-containing compound therein. This embodiment of the invention includes a seam 24 that allows application
15 of the gasket to a cable even when the cable (not shown) cannot easily be "broken." According to this aspect of the invention the seam includes portions 26 that run collinear with the underlying cable. The collinear sections are separated by seam portions 28 that run at an
20 angle to the first portions, preferably perpendicular to the first portions.

As indicated by the arrows in Figs. 8a and 8b, a force is applied to the faces of the gasket, thereby placing a force on adjoining faces of the seam sections
25 28. Such force may be applied by, for example, tightening of a nut against one face while holding the other face stationary with a raised face of an adjoining piece of equipment. Accordingly, even though the gasket contains a seam, the seam will prevent leaks along the
30 seam at the compressed seam sections 28. Moreover, during cooling, the seam sections 28 will remain in close contact as a result of the low coefficient of expansion fill material in the gasket.

In preferred embodiments, it may be desirable
35 to modify the contour of the seal wall(s) to prevent buckling of the inner wall.

1. Example

Various seam configurations were investigated.

Results from an experiment in which glass bead-based seals having straight "step" and no seam are listed in
5 Table 4. These data indicate first of all that the performance of the glass bead-based seal, regardless of the type of seam, is superior to that of solid rubber seals. The effectiveness of the "step" seam could not be
10 determined because no glass bead-based seal failures occurred. The purpose of the "step" seam was to take advantage of the pressure exerted on the seal to compress the portion of the seam that is normal to the direction of the applied force. The simplest use of this principle is the "step" seam used herein. However, there are
15 numerous other ways of accomplishing this.

Table 4
Thermal Cycling of Vitroseal Seam Sealing Study
 (2" Polypropylene fixtures, 0.67" OD FO cable)

<u>Sample</u>	<u>Type of Seal</u>	<u>No. of Cycles</u>	<u>Type of Failure</u>
<u>No Seam:</u>			
13698-2a	Glass Bead Filled	151 (ongoing)	
13698-2b	Glass Bead Filled	151 (ongoing)	
13698-2c	Glass Bead Filled	119 (ongoing)	
13698-1a	Solid Rubber	3 (failed)	Leak along cable
13698-1b	Solid Rubber	6 (failed)	Leak along cable
13698-1c	Solid Rubber	12 (failed)	Leak along cable
<u>Straight Seam:</u>			
13698-3a	Glass Bead Filled	110 (ongoing)	
13698-3b	Glass Bead Filled	72 (ongoing)	
13698-3c	Glass Bead Filled	150 (ongoing)	
13698-8a	Solid Rubber	3 (failed)	Leak along seam
13698-8b	Solid Rubber	3 (failed)	Leak along seam
13698-8c	Solid Rubber	3 (failed)	Leak along seam
<u>Step Seam:</u>			
13698-4a	Glass Bead Filled	151 (ongoing)	
13698-4b	Glass Bead Filled	151 (ongoing)	
13698-4c	Glass Bead Filled	151 (ongoing)	
13698-5a	Solid Rubber	106 (failed)	Leak around cable
13698-5b	Solid Rubber	48 (failed)	Leak around cable
13698-5c	Solid Rubber	154 (ongoing)	

Although no apparent problems were observed during installation of seals having the "step" seam, it is believed that the longitudinal walls of the seam might offer some additional resistance to compression and, thus, increase the force necessary for installation. To solve this potential problem, a "saw tooth" seam configuration may sometimes be desirable. This type of seam design will allow the seal walls to collapse more easily than straight walls and, as a result, reduce the force required for installation.

B. Cable Enclosure

Figs. 9a and 9b illustrate a gasket and associated cable enclosure. The cable enclosure in Figs. 9a and 9b may be used with, for example, fiber optic, coaxial, or copper cables.

As shown in Fig. 9a, the gasket 31b is made from separable pieces 30 and 32. The two pieces form halves of a circle with one or more cable and stud passageways 34. The gasket is made according to one of the embodiments using a body of, for example, silicon rubber filled with a low coefficient of thermal expansion filler.

As shown in Fig. 9b, the two pieces of the gasket are brought together in a reenterable cable enclosure 36 that is used, for example, in protection of cable splices (not shown). The cable enclosure has recessed portions 38 that are formed to receive the gasket 31. Extending from the recessed portion are studs 40. The gasket is placed in the recessed region with the studs passing through stud passageways 42.

Fig. 9c illustrates an alternative embodiment of the invention in which the face of the gasket on one or more of the surfaces to be sealed is provided with a furrowed surface 43. In addition, the surfaces of the gasket portions that are to be joined are provided with similar furrowed surfaces 45. Such surfaces provide

pressure points that will provide more effective sealing in many applications.

Fig. 9d illustrates another alternative embodiment of a two piece gasket assembly. According to
5 this embodiment, a flat slab of gasket material 47 is provided between the two furrowed gasket portions 45.

Fig. 9e illustrates a two piece gasket with a lateral face 28 analogous in function to the embodiment shown in Fig. 8a. When pressure is exerted on the faces
10 28 the leak path formed by the seal break will be effectively blocked.

A pressure plate 44 having the same general shape as the gasket is then placed over the studs adjacent the gasket. The pressure plate is then
15 compressed against the body 36 using nuts 46, optionally abutting springs 48. When the sealing system is formed around one or more cables, it will provide an environmentally secure enclosure that will not be prone to failure during low temperature operation.

20 Figs. 10a and 10b illustrate an in-line enclosure for cable splices and the like. Fig. 10a shows the device in cross section without a cable, while Fig. 10b shows the device with an installed cable. Similar to the embodiment illustrated above, the
25 enclosure includes a body 36. A gasket 31 is held tightly against the body by way of a washer 44 and a threaded plunger 50 that engages the body, forcing the gasket into the recessed region therein.

Figs. 11a and 11b illustrate a butt splice
30 closure using gaskets of the present invention. Fig. 11a illustrates the device in exploded view, while Fig. 11b illustrates the device in a partially assembled configuration. In this case, two or more cables 60 enter the enclosure via a cap assembly 62 having cable
35 apertures therein. The cap assembly and the body 64 have retaining walls 64 that are spaced apart a selected distance when the cap is placed on the body. A seal 66,

like those described above, is placed around the body between the retaining walls and held thereto with a steel band 68. An additional low temperature gasket 70 engages the cap and the cable inserted therein using a washer 74 and plunger 76.

C. Hybrid Seal System

Figs. 12a and 12b illustrate a hybrid sealing system similar to the above described embodiments having two abutting sections. Fig. 12a illustrates the seal system in cross section, while Fig. 12b is a perspective view. In this particular embodiment, a ring of soft, compliant material 80 is placed in a ridge 82 around a central portion of the seal. This soft material will seal the walls of adjoining surfaces having minor imperfections that cannot easily be sealed by hard rubber gaskets. Such minor imperfections may arise from scratches, molding imperfections, or other similar defects.

Preferably, the material 82 is a soft, pliable, and tacky material. For example, it is preferred that the cone penetrates to between about 80 to 360×10^{-1} mm. In one embodiment, the cone penetration value is between about 250 and 360. Suitable materials include gels (thermoset or thermoplastic, such as those described in copending U.S. application Serial No. 07/802,950, incorporated herein by reference for all purposes), mastics, putties, pastes, polyisobutylene (crosslinked or not crosslinked), high viscosity greases, highly extended rubbers, partially crosslinked rubbers or pressure sensitive adhesives.

Figs. 13a and 13b provide cutaway and isometric views of a gasket similarly equipped with a ring of soft material 82 with a gasket of a cylindrical configuration.

1. Example

To test the effectiveness of hybrid seals, two sets of samples were prepared. The first set consisted of a sandwich configuration with a mastic ring compressed between two split tubular seals with glass beads.
5 Control samples were also tested which consisted of two split tubular seals but, no mastic. The seams of the two seals were staggered so as to simulate a "step" seam. Although initially this was done to avoid making a mold
10 for the rather complex shape, it turned out to be a good approach for making hybrid seals in a simple manner.

The results in Table 5 indicate that the use of mastic has improved the pressure retention ability of these samples. After 151 cycles the mastic-containing
15 samples retained about 90% of their initial pressure, while those containing no mastic retained only 70%. This seems to indicate that the presence of micro imperfections may cause very slow leaks which cannot be easily detected. As seen by the data listed in Table 5,
20 no failures occurred as yet in either set of samples. Solid rubber seals containing a mastic ring, on the other hand, failed overnight (i.e., in less than 2 cycles).

Table 5
Thermal Cycling of Hybrid Seals
(Polypropylene fixtures, FO cable)

<u>Sample</u>	<u>No. of Cycles</u>	<u>Internal Pressure¹</u>	<u>Type of Failure</u>
<u>Blass Bead-Based Seals with Mastic:</u>			
13698-1-6a	151 (ongoing)	4.5 psi	
13698-1-6b	151 (ongoing)	4.7 psi	
13698-1-6c	151 (ongoing)	4.3 psi	
<u>Glass Bead-Based Seals without Mastic:</u>			
13698-1-7a	143 (ongoing)	3.5 psi	
13698-1-7b	143 (ongoing)	3.4 psi	
13698-1-7c	143 (ongoing)	3.6 psi	
<u>Solid Silicone Rubber with Mastic:</u>			
13452-1a	2 (failed)	0 psi	Leak along cable ²
13452-1b	2 (failed)	0 psi	Leak along cable ²
13452-1c	2 (failed)	0 psi	Leak along cable ²

1. Initial pressure was 5 psi.
2. Cable region that was in contact with rubber was highly deformed.

D. Differential Expansion Seal

Figs 14a and 14b illustrate an alternative embodiment of the invention in which rings 90 surround a portion of the outer perimeter of the gasket. Fig. 14a illustrates the invention at room temperature, while Fig. 14b illustrates the invention at a lower temperature, such as -40°C. Rings 90 have a higher coefficient of thermal expansion than either the body or the fill material. Accordingly, when the gasket is cooled, the walls of the gasket are forced outwards due to the volume displacement of the gasket within the rings. The amount of displacement can easily be adjusted by adjusting the amount of the size of the rings 90 and relative coefficient of thermal expansion of the materials. Of course, in this embodiment, the ring need only have a lower coefficient of thermal expansion than the body and not necessarily a low coefficient of thermal expansion.

20

1. Example

Rings according to Fig. 14 were tested in aluminum test fixtures and pressurized to 5 psi. The gaskets were thermally cycled from -40°C to +60°C. Gaskets with low coefficient thermal expansion fill failed only after more than 50 cycles, while 100% silicone rubber gaskets failed after 15 cycles.

E. Wraparound Sealing Device

Figs. 15a to 15d illustrate additional embodiments of the invention. The versions shown in Fig. 15a to 15d can be used to produce thin-walled devices for either front access only (e.g., 15a) or for applications in which there is access from both sides. In the latter case, it is possible to pre-adjust either the inner or outer tubular gasket with a threaded ring. This feature could be useful in situations where there is a need to extend the sealing range of either gasket.

The embodiments shown in Fig. 15 are referred to herein as thin walled enclosures.

In Fig. 15a front access is provided. Gasket displacement is varied by piston 1503 or gasket length.
5 In Fig. 15b access is provided on both sides. The outer gasket can be preadjusted with the threaded ring 1501. The outer threaded cap 1502 can compress both gaskets. In Fig. 15e access is provided from both sides and the outer gasket is compressed by threaded ring 1501 while
10 the threaded cap 1502 compresses the inner gasket. Fig. 15d illustrates a device with access from either side, and compression is provided by caps 1504.

The diagrams in Fig. 16a to 16d show some examples of devices in which pistons and tubular gaskets
15 are placed on both sides of the main body. As in Fig. 15, there are many variations that can be made depending on the type of application. The embodiments shown in Fig. 16 are referred to herein as inside/outside gasketed wall embodiments. The gaskets can be compressed
20 independently by threaded caps or rings, or they can be compressed simultaneously by one threaded cap. Fig. 16a illustrates a device with front access where gasket displacement is varied by piston 1601 or gasket length. Fig. 16b illustrates a device where access is from both
25 sides and where the cap 1603 compresses the outer gasket of the cap 1605. Fig. 16c illustrates a device with front access where the gaskets are independently compressed by threaded cap 1607 or ring 1609. Fig. 16d illustrates a device with front access wherein the inner
30 gasket is adjusted with ring 1611 and both gaskets are compressed by cap 1615.

Examples of another version are shown in Figs. 17a to 17c. These devices make use of only one piston to compress a tubular inner gasket. Sealing
35 around the port or outer region is accomplished by the use of a flat gasket which is compressed by either an independent threaded ring or by the pulling action of

the threaded cap on the main body. The embodiments in Fig. 17 are referred to herein as single piston embodiments.

Fig. 17a illustrates a seal system in which front access is provided and both gaskets are individually compressed. Fig. 17b illustrates a seal system in which front access is provided and both gaskets are compressed. Fig. 17b illustrates a device with access from both sides and a flange 1701 assists in compressing the outer gasket. Fig. 17c illustrates a device with access from both sides in which both gaskets are compressed at the same time by cap 1703.

One of the most important advantages over traditional gaskets and "O" rings is the fact that the ratio of length of gasket to piston displacement can be varied over a wide range, thus providing a wide sealing range capability. This is illustrated by Figs. 18a-18c. Fig. 18a provides the same compression on both gaskets while Figs. 18b and 18c show devices wherein one gasket is preferentially compressed by ring 1801.

Seam sealing can be accomplished by allowing both gaskets to come in contact with each other through an opening along the seam. This is illustrated by the diagrams in Figs 19a to 19d, which embodiments are referred to as seam sealing embodiments.

F. EMI/RFI Protection

The sealing of watertight enclosures for protection of sensitive electronic controls with EMI/RFI protection is difficult to achieve with prior methods. A solution to the problem is to encapsulate conductive microspheres or liquids within an elastomeric sleeve so that sealing and EMI/RFI protection can be achieved in a simple step. This approach lends itself to the production of all types of gaskets or sealing devices (O-rings, grommets, bushes, plugs, boots and flat or tubular gaskets).

Materials suitable for this application are electrically conductive solid and liquid materials (e.g., metal and metal alloys, graphite, carbon black, as well as conductive synthetic polymers, gels and liquids).

5

IV. Conclusion

The present invention provides a greatly improved method and device for forming seals in low temperature applications, and especially where thermal cycling is expected. The above description is illustrative and not restrictive. Many variations of the invention will become apparent to those of skill in the art upon review of this disclosure. Merely by way of example the various components have the invention have been illustrated with respect to various specific materials, but the scope of the invention is not so limited. The scope of the invention should, therefore, be determined not with reference to the above description, but instead should be determined with reference to the appended claims along with their full scope of equivalents.

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WHAT IS CLAIMED IS:

1. A sealing member comprising:
an elastomeric body defining an internal
5 cavity, said elastomeric body formed to engage at least
two surfaces to be sealed against leakage, said
elastomeric body having a first coefficient of thermal
expansion; and
a compliant filling material, said compliant
10 filling material selected from the group consisting of
materials with a coefficient of thermal expansion less
than said first coefficient of thermal expansion,
materials that expand with lowering temperature on at
least a portion of a temperature range of interest, and
15 combinations thereof.
2. A sealing member as recited in claim 1 wherein
said filling material substantially fills more than 50%
of the internal volume of said body.
20
3. A sealing member as recited in claim 1 wherein
said filling material fills more than 70% of the internal
volume of said body.
- 25 4. A sealing member as recited in claim 1 wherein
said fill material has a coefficient of thermal expansion
of less than 200×10^{-6} m/m/°K.
5. A sealing member as recited in claim 1 wherein
30 said fill material has a coefficient of thermal expansion
of less than 5×10^{-6} m/m/°K.
6. A sealing member as recited in claim 1 wherein
said body is a thermoset elastomer.
35
7. A sealing member as recited in claim 6 wherein
said body is silicone rubber.

8. A sealing member as recited in claim 1 wherein said filling material comprises a material selected from the group consisting of glass, graphite, carbon, ceramic, epoxy, mica, and combinations thereof.

5

9. A sealing member as recited in claim 1 wherein said filling material comprises finely divided glass.

10. A sealing member as recited in claim 1 wherein said filling material comprises finely divided solid particles.

11. A sealing member as recited in claim 10 wherein said solid particles have an average diameter of less than 1000 microns.

15

12. A sealing member as recited in claim 10 wherein said solid particles have an average diameter of less than 500 microns.

20

13. A sealing member as recited in claim 10 wherein said particles are closely packed whereby a void volume in said cavity is less than about 50%.

25

14. A sealing member as recited in claim 10 wherein interstices between said particles are unfilled.

15. A sealing member as recited in claim 1 wherein a compressive force generated between two said sealing member and adjoining surfaces is substantially greater when a temperature of said gasket is lowered from 20°C to -40°C than a sealing member made entirely of a material of fabrication of said body.

30

16. A sealing member as recited in claim 1 wherein said filling material expands at temperatures below 4°C.

35

17. A sealing member as recited in claim 16 wherein said filling material comprises a material selected from the group consisting of water, hydrogels, water swellable polymers, and combinations thereof.

5

18. A sealing member as recited in claim 16 wherein said filling material is a hydrogel selected from the group consisting of polyethylene oxide, polyacrylic and polymethacrylic acids, polyacrylic and polymethacrylic esters, polyhydroxyalkyl metacrylates or acrylates, polyacrylamide or polymetacrylamide, poly-n-isopropylacrylamide, poly-n-vinyl-1-2-pyrrolidinone, crosslinked polyvinyl alcohol, polyvinyl propynol, polyethylene oxide, carboxymethyl or hydroxyethyl cellulose, hydroxypropyl methylcellulose, crosslinked polypropenoic acid, polyelectrolyte complexes, charge polymers carrying ionizable groups, and mixtures thereof.

19. A sealing member as recited in claim 1 wherein said body is cylindrically shaped and contains a longitudinal aperture therethrough.

20. A sealing member as recited in claim 19 wherein said cylindrically shaped body is split along a side thereof, said split comprising at least a substantially lateral segment therein.

21. A sealing member as recited in claim 1 further comprising a mastic adjacent one of said surfaces to be sealed.

22. A seal as recited in claim 21 wherein said mastic is selected from the group consisting of thermoset gels, thermoplastic gels, mastics, putties, pastes, polyisobutylene, grease, extended rubber, and combinations thereof.

23. A sealing member as recited in claim 1 wherein said filling material comprises a conductor, whereby said seal provides electromagnetic and radio frequency protection.

5

24. A sealing member as recited in claim 1 further comprising a seal carrier selected from the group consisting of thin walled enclosures, inside/outside gasketed wall enclosures, single piston enclosures, and seam sealing enclosures.

10

25. A seal comprising at least a ring of material having a first coefficient of thermal expansion, said ring surrounding an internal body, a wall of said body engageable with a surface to be sealed, said ring of material having a coefficient of thermal expansion less than said body, whereby upon cooling said ring contracts to forcibly engage said wall with said surface to be sealed.

15

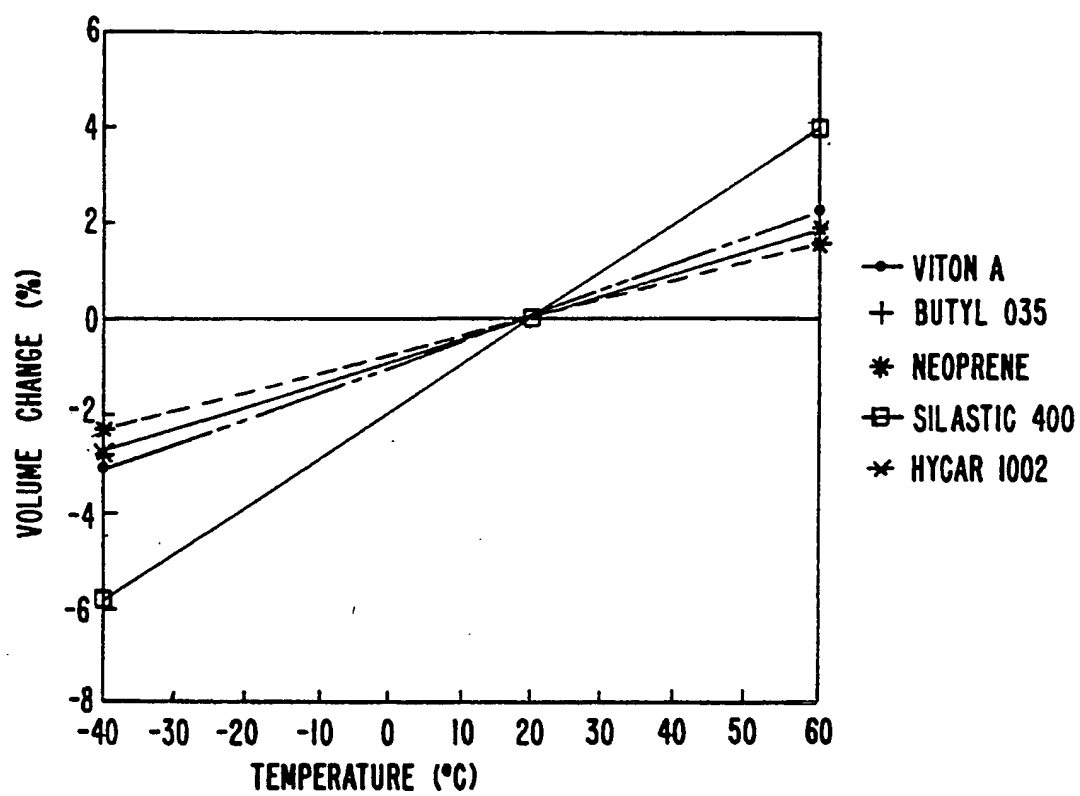
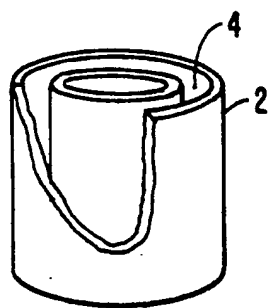
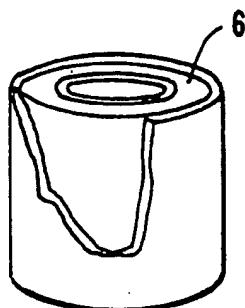
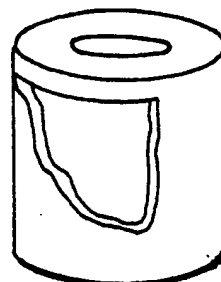
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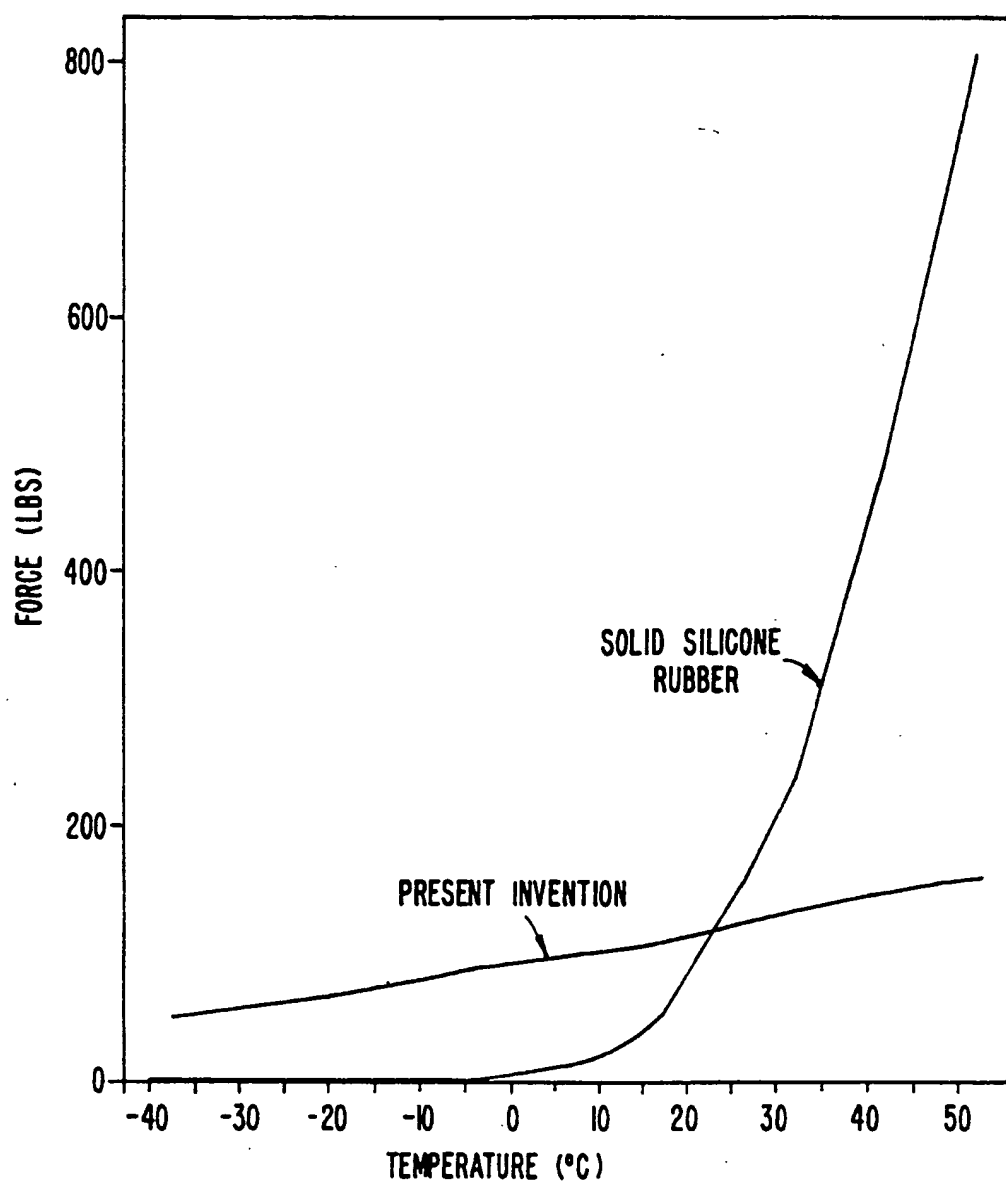
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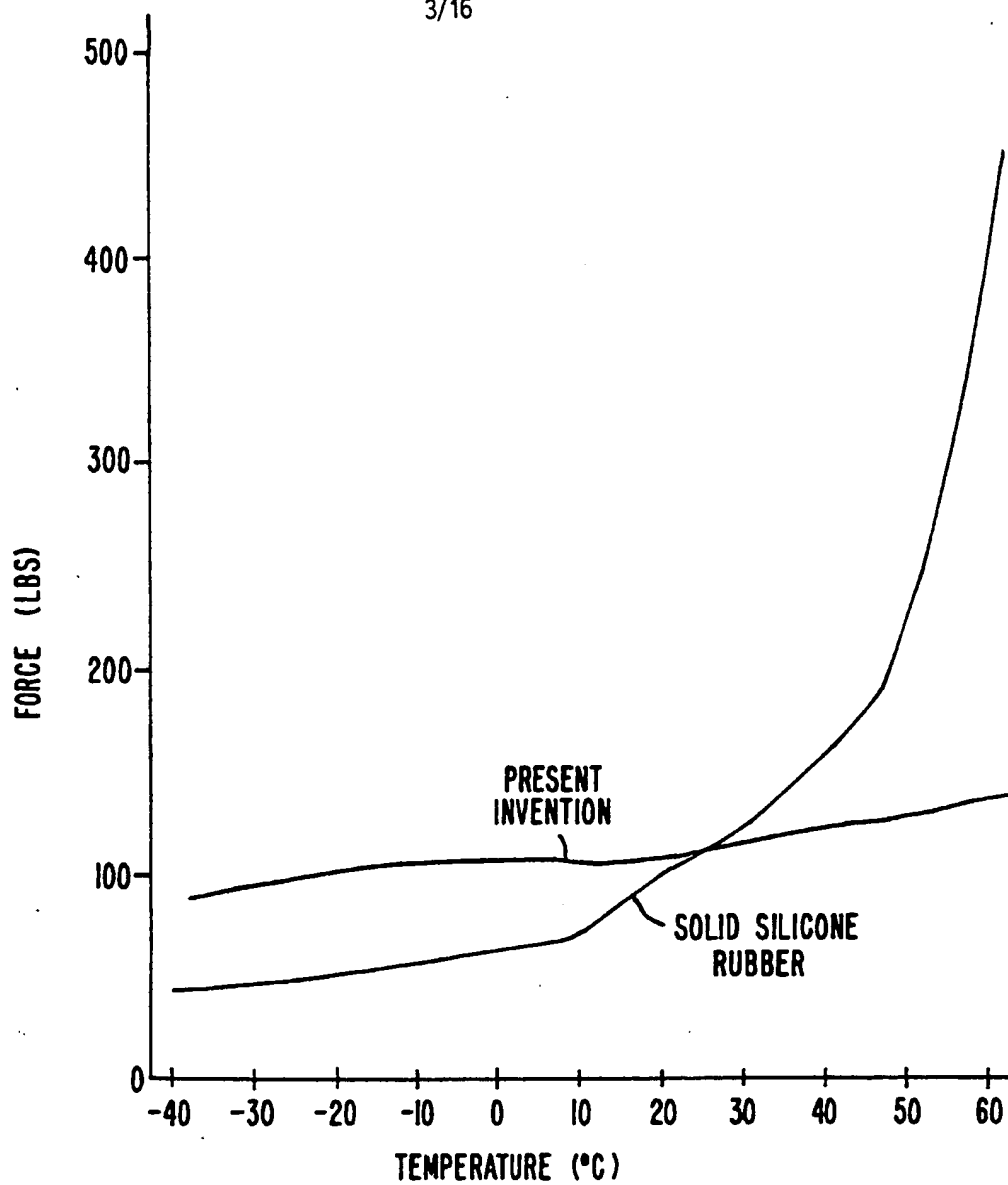
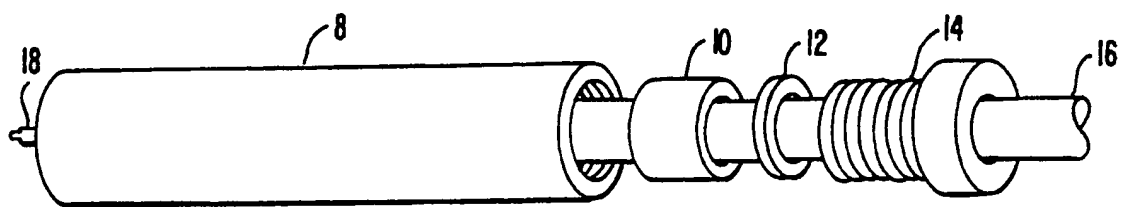
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**FIG. 1.****FIG. 2A.****FIG. 2B.****FIG. 2C.**

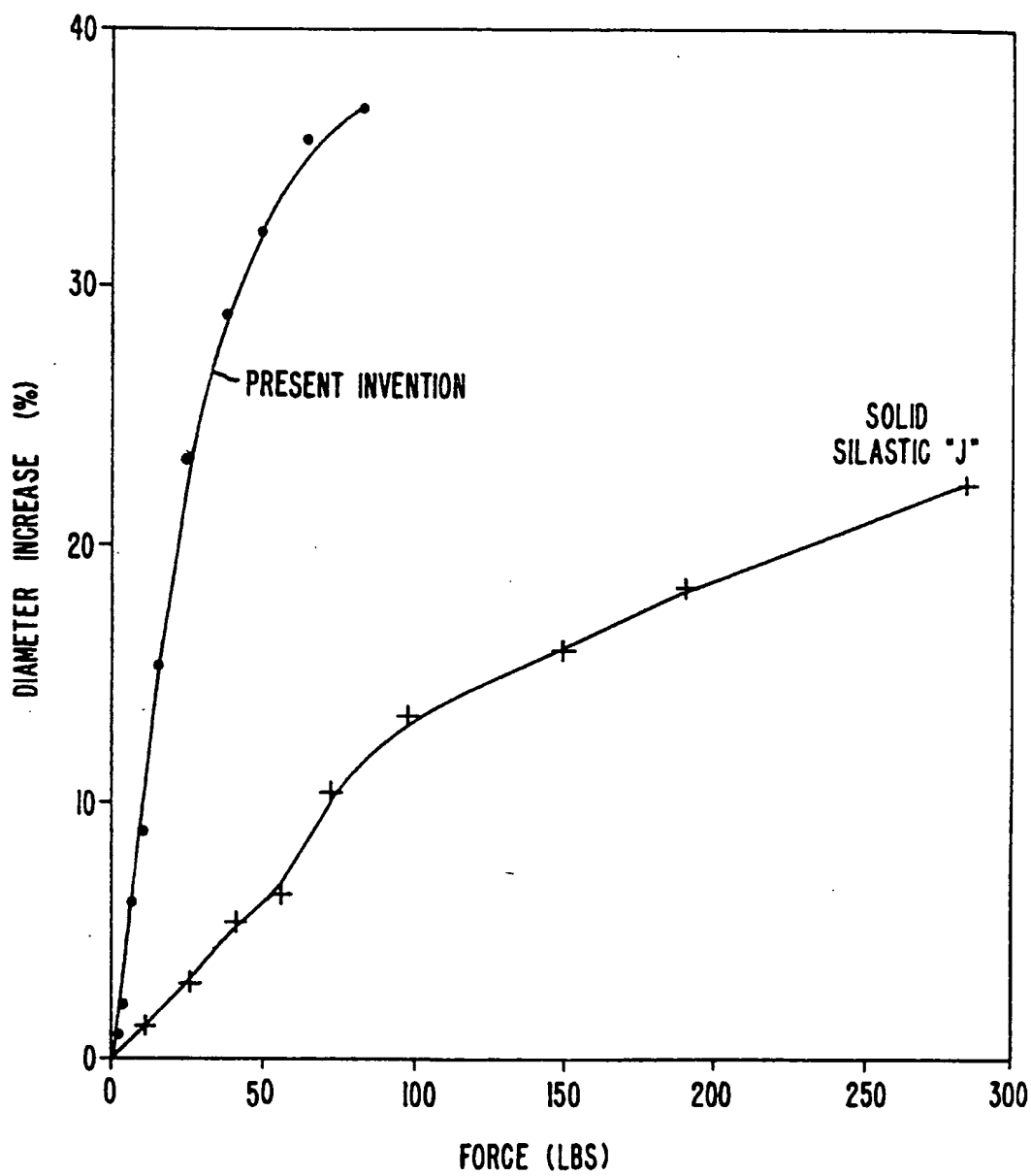
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**FIG. 3A.**

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**FIG. 3B.****FIG. 4.**

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**FIG. 3C.**

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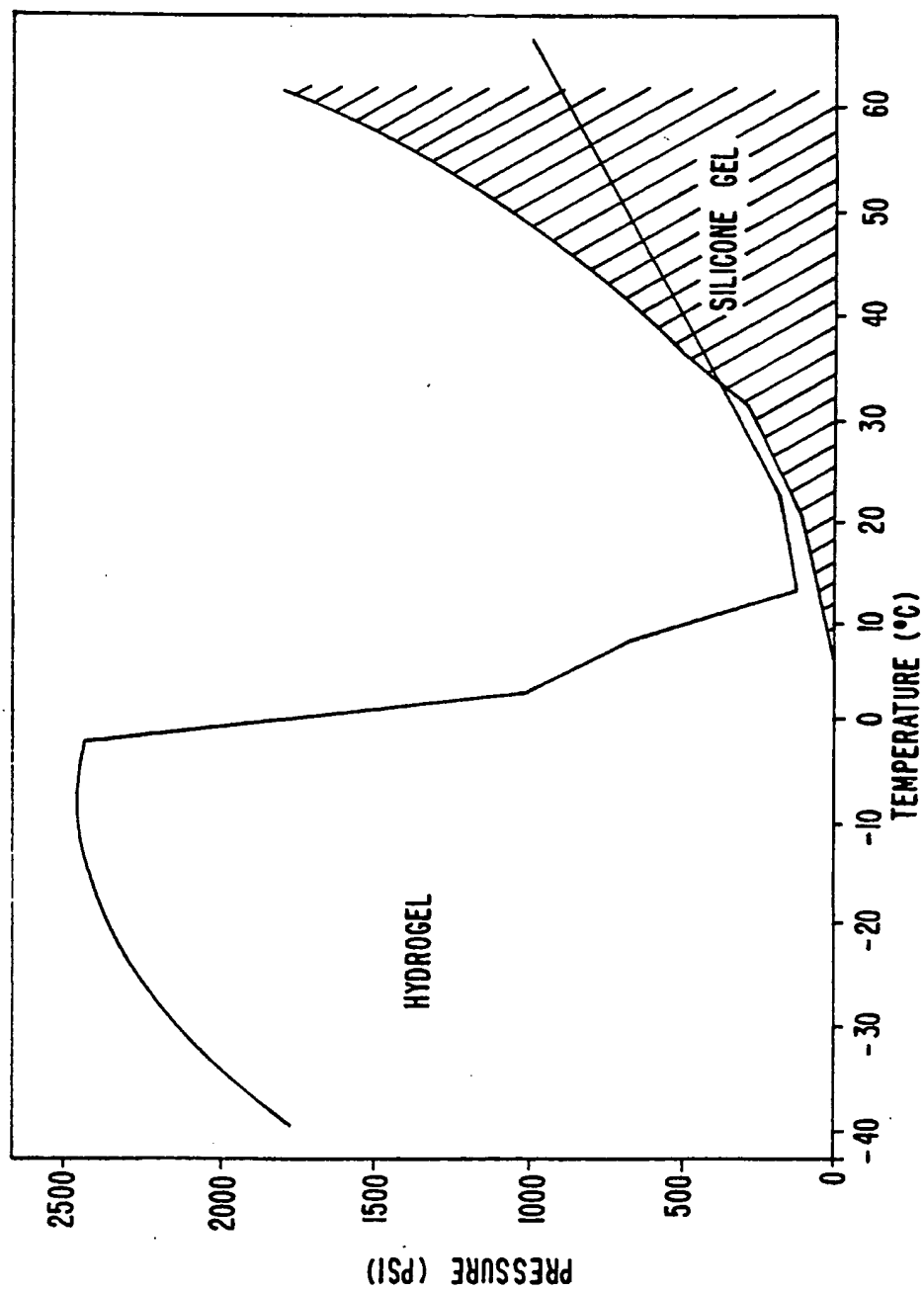
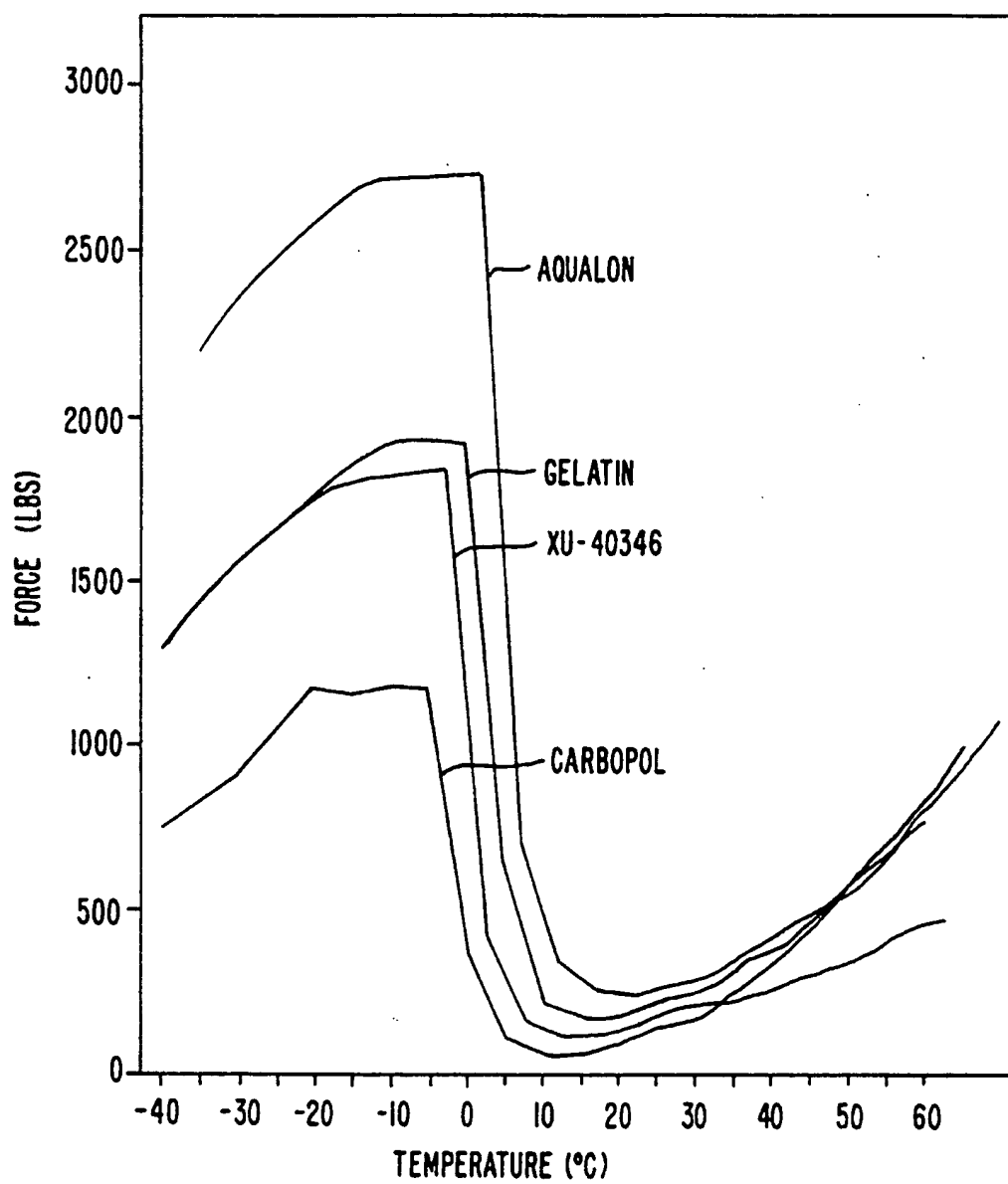


FIG. 5.

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**FIG. 6.**

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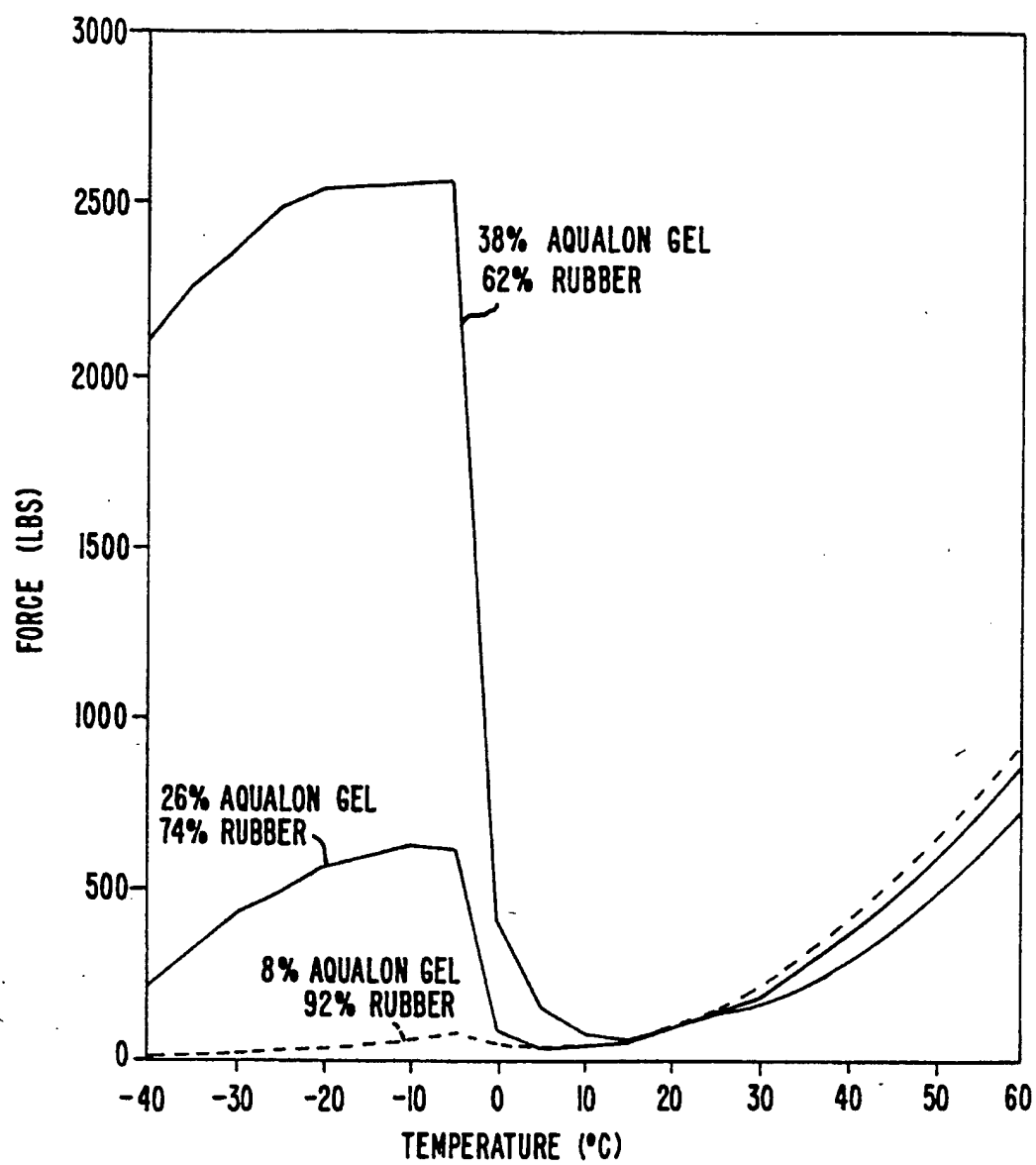


FIG. 7.

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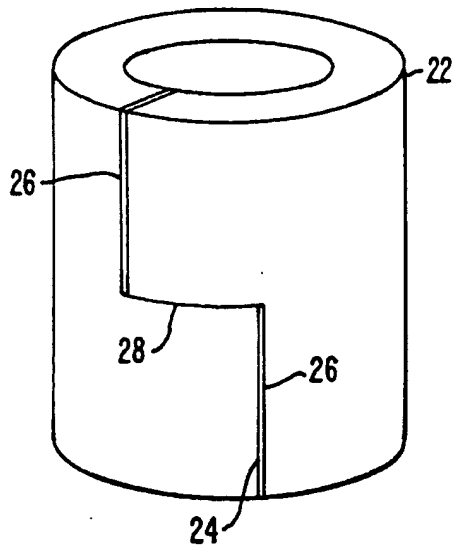


FIG. 8A.

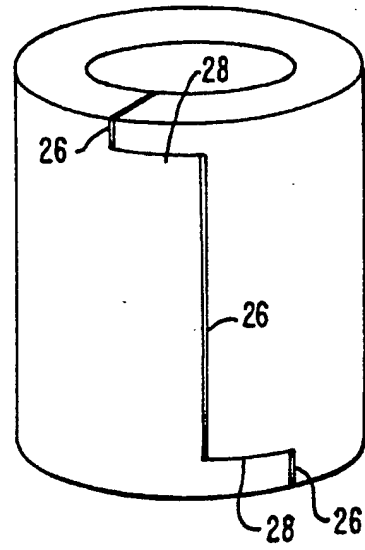


FIG. 8B.

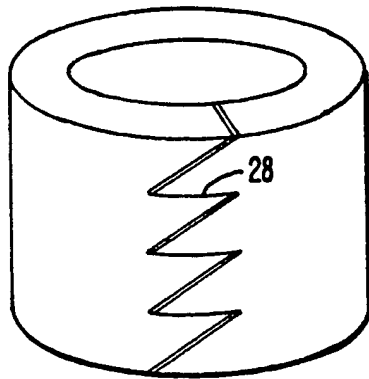


FIG. 8C.

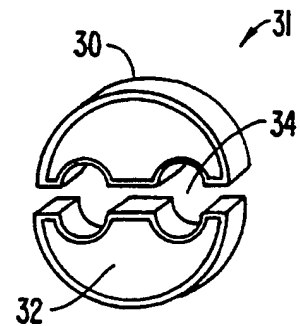
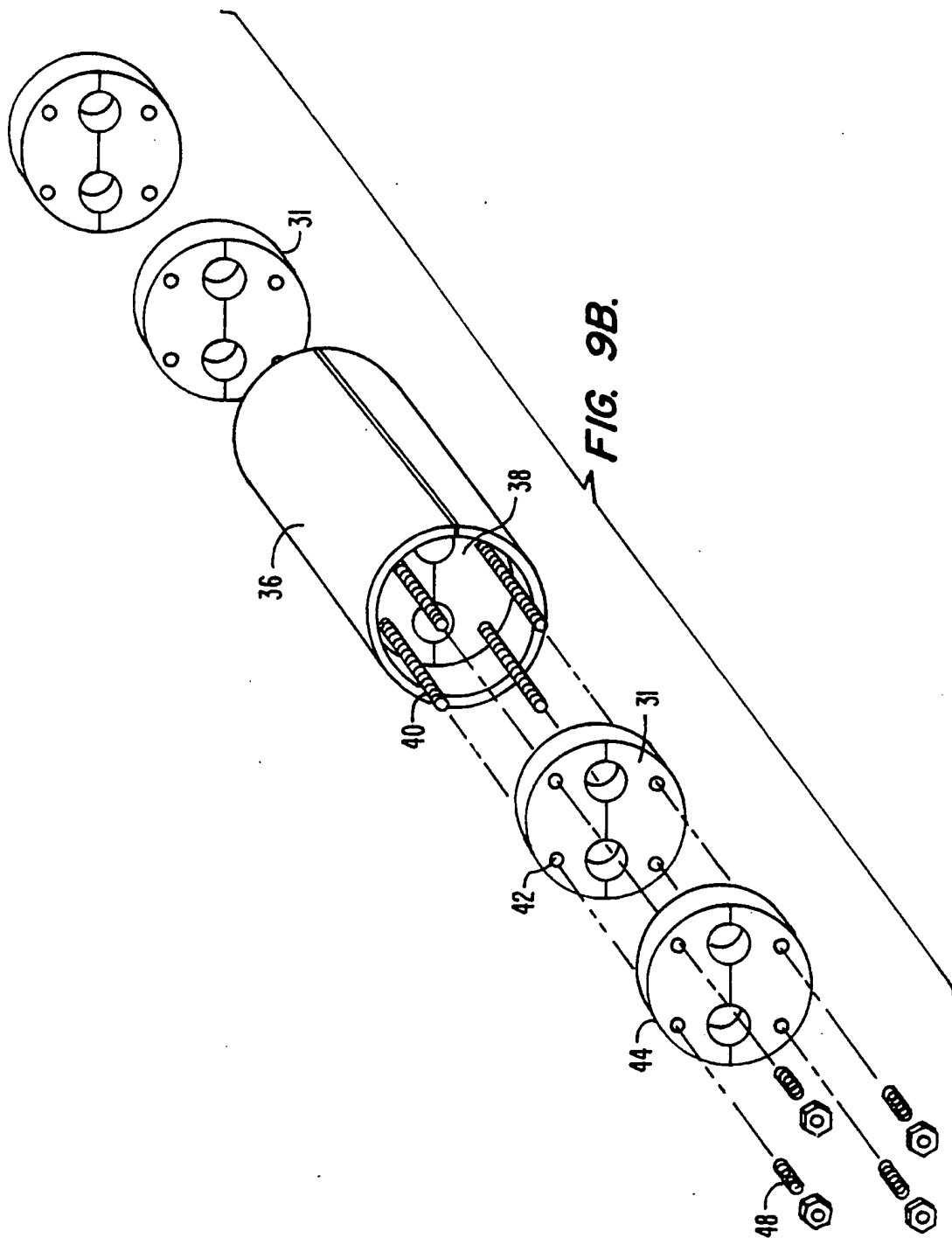


FIG. 9A.

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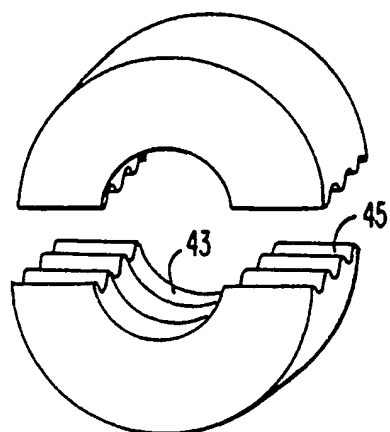


FIG. 9C.

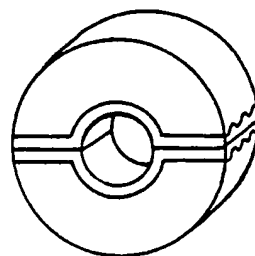
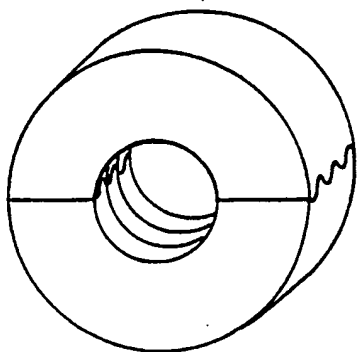


FIG. 9D.

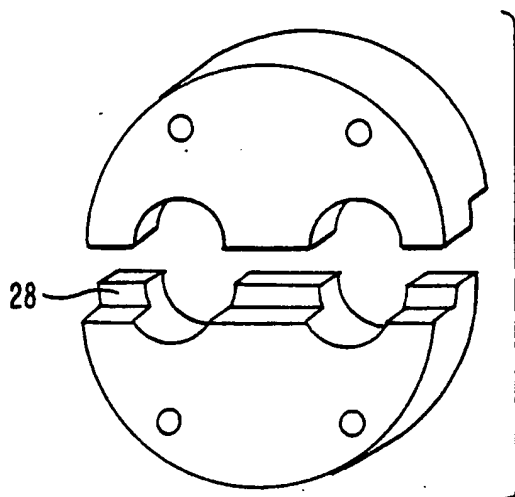
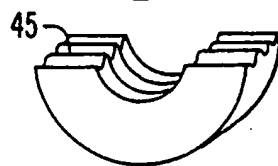
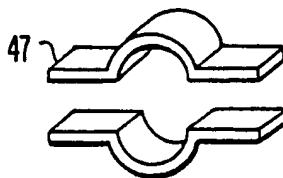


FIG. 9E.

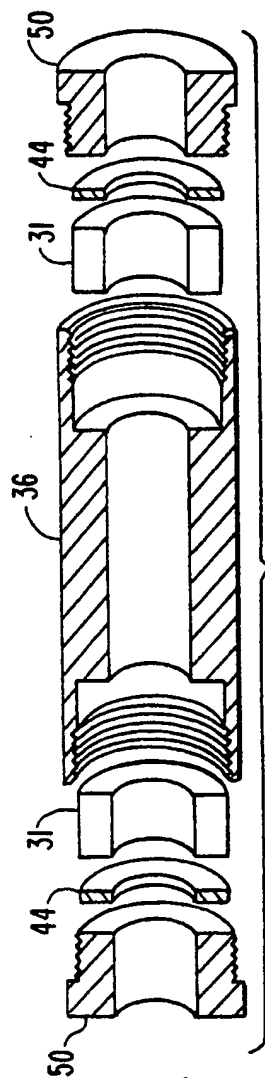


FIG. 10A.

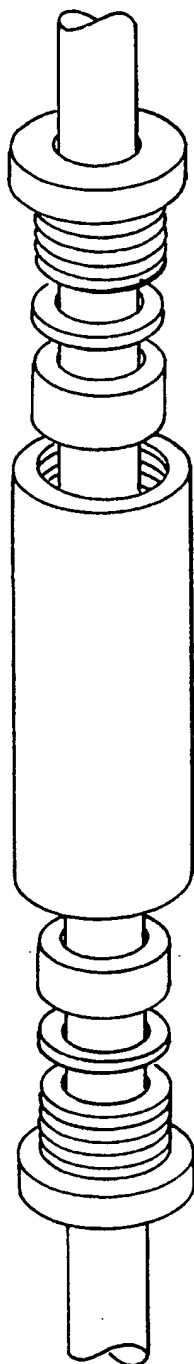


FIG. 10B.

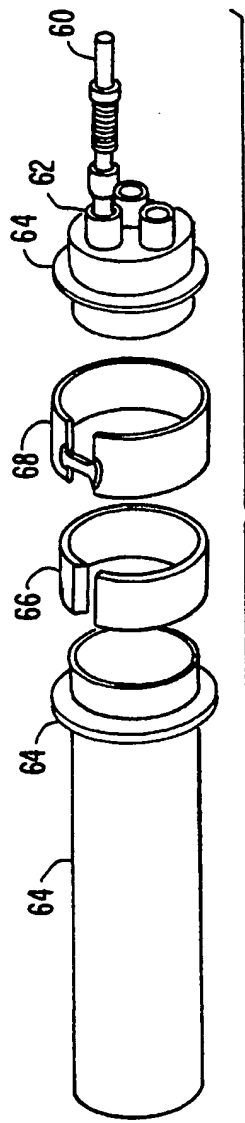


FIG. 11A.

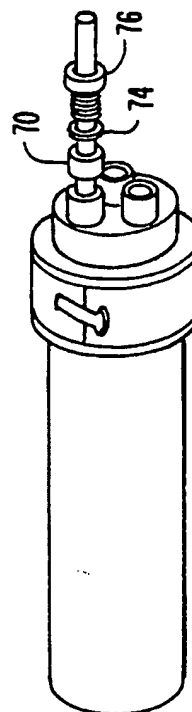


FIG. 11B.

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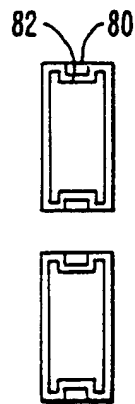


FIG. 12A.

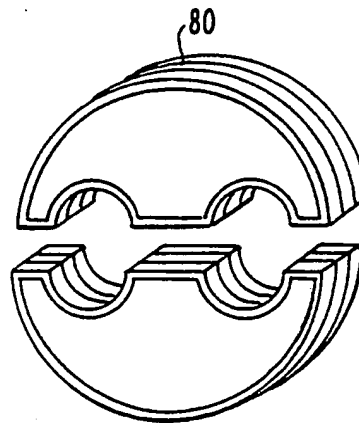


FIG. 12B.

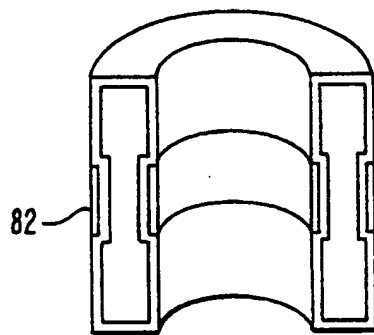


FIG. 13A.

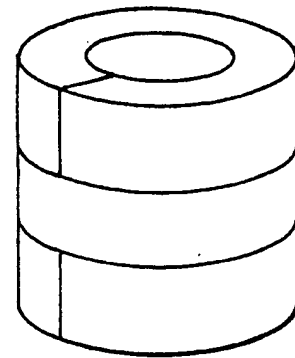


FIG. 13B.

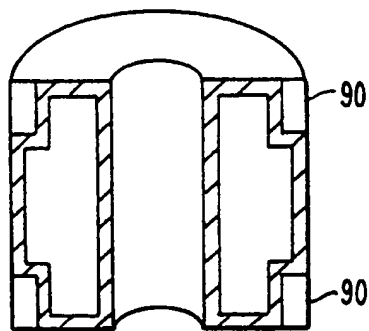


FIG. 14A.

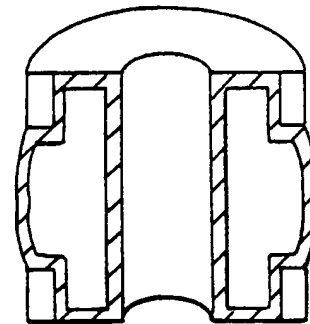


FIG. 14B.

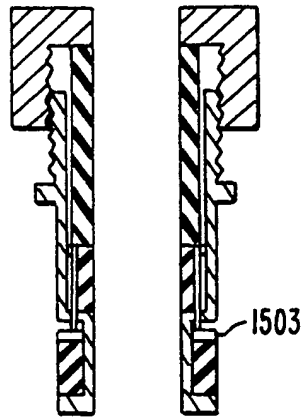


FIG. 15A.

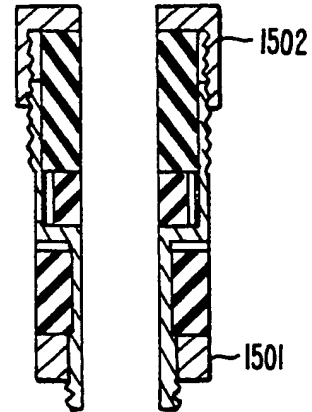


FIG. 15B.

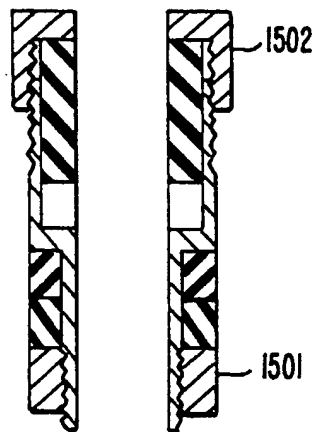


FIG. 15C.

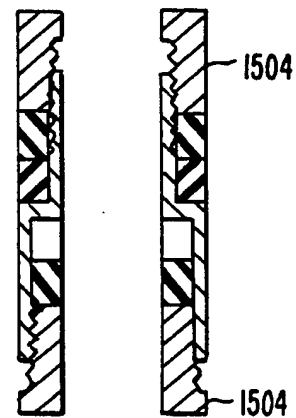


FIG. 15D.

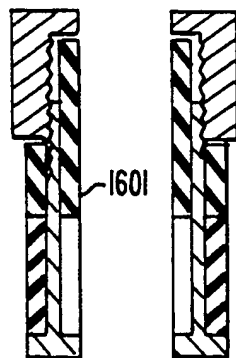


FIG. 16A.

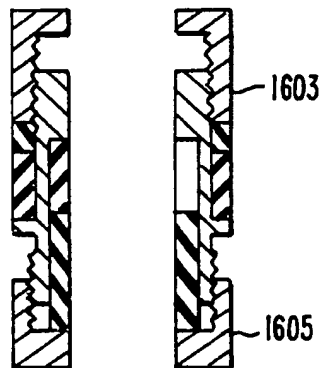
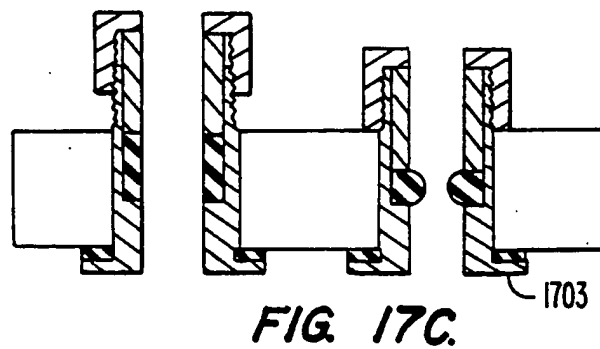
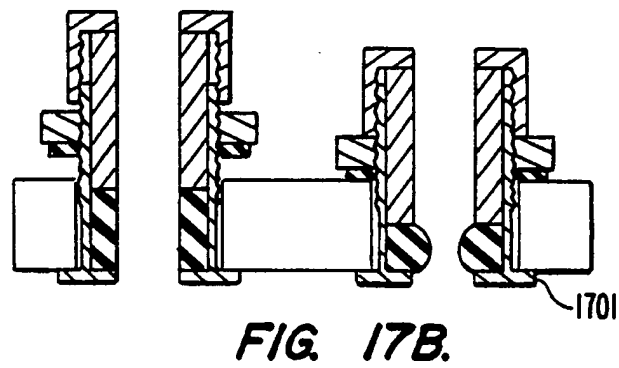
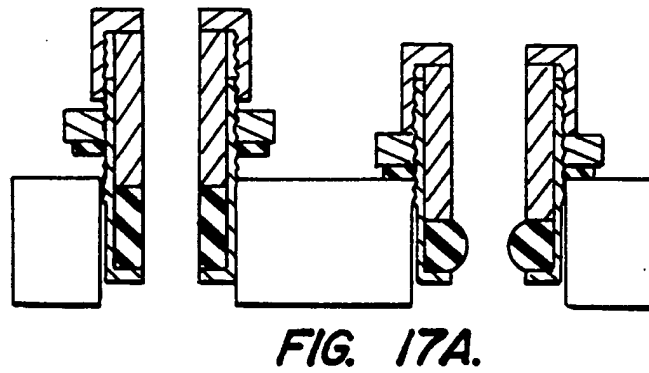
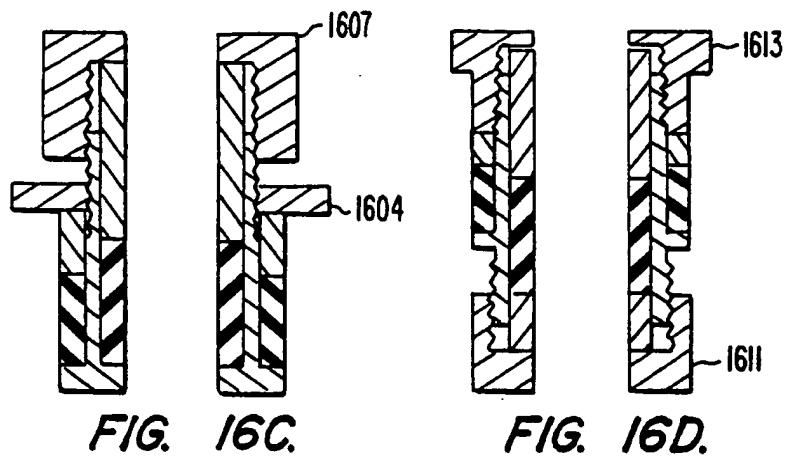


FIG. 16B.



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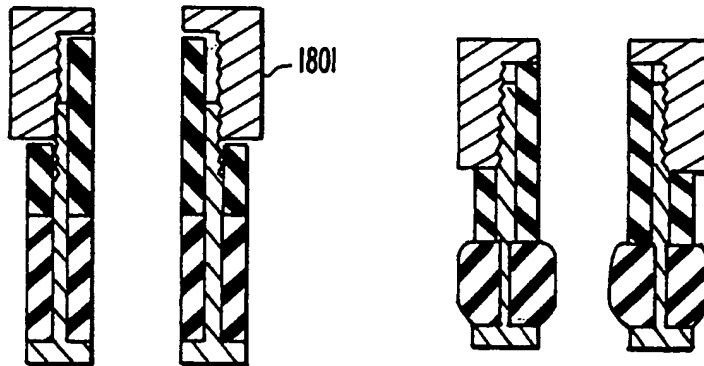


FIG. 18A.

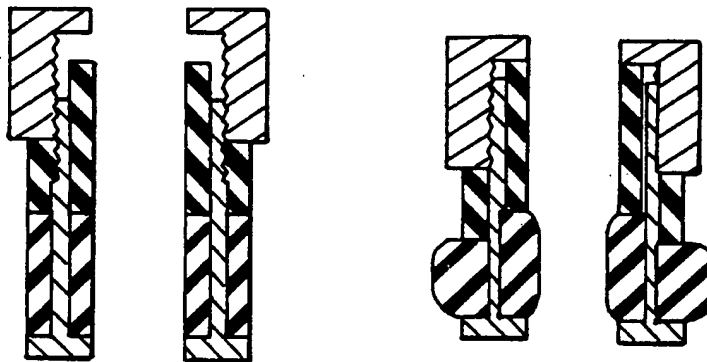


FIG. 18B.

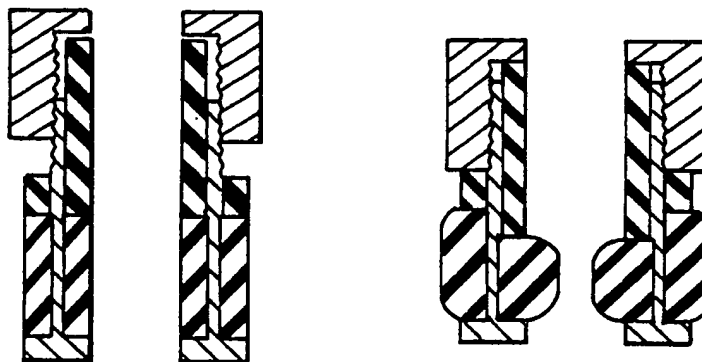


FIG. 18C.

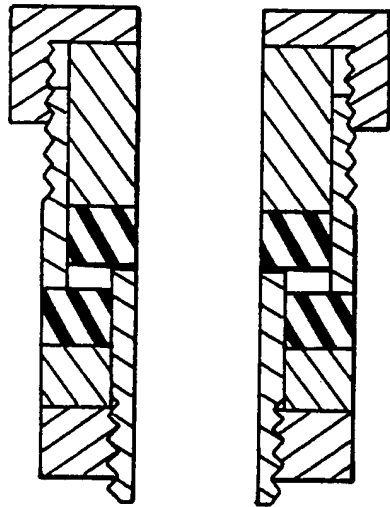


FIG. 19A.

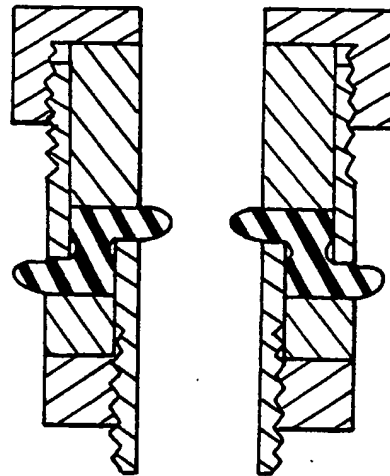


FIG. 19B.

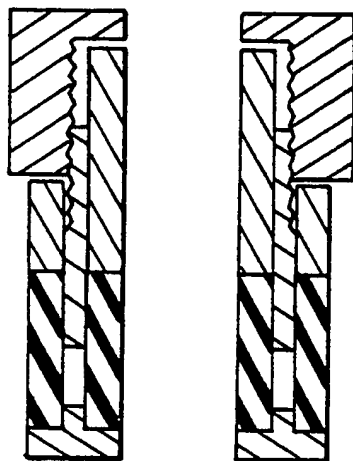


FIG. 19C.

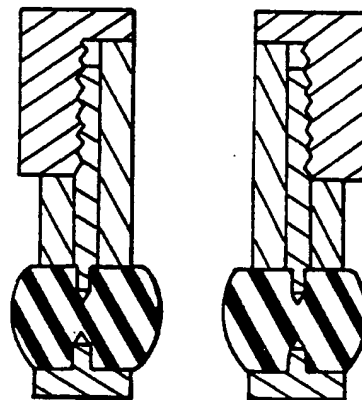


FIG. 19D.

INTERNATIONAL SEARCH REPORT

International Application No

PCT/US 93/07416

A. CLASSIFICATION OF SUBJECT MATTER
 IPC 5 F16J15/16 H02G3/06

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC 5 F16J H02G

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	DE,C,30 18 215 (FICHTEL & SACHS) 19 November 1981 see the whole document	1,15-17, 19,25
A	--- ADVANCES IN CRYOGENIC ENGINEERING - PROCEEDINGS OF THE 1961 CONF. UNIVERSITY MICHIGAN vol. 7 , 1962 , NEW YORK pages 556 - 561 S.E. LOGAN 'temperature-energized seal for liquid hydrogen' -----	1

☐ Further documents are listed in the continuation of box C.

☒ Patent family members are listed in annex.

* Special categories of cited documents :

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- *L* document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)
- *O* document referring to an oral disclosure, use, exhibition or other means
- *P* document published prior to the international filing date but later than the priority date claimed

- *T* later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
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- *Y* document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.
- * & * document member of the same patent family

Date of the actual completion of the international search

11 November 1993

Date of mailing of the international search report

30. 11. 93

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 Fax (+ 31-70) 340-3016

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LEGER, M

information on patent family members

PCT/US 93/07416

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Patent family member(s)

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DE-C-3018215

19-11-81

DE-A, C 3018215

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